7 GAS CHROMATOGRAPHS

A. The GC septum is changed during monthly instrument calibration or as needed.

B. The glass liners are changed or cleaned as needed.

C. All gas cylinders used are checked at least once daily and changed at a minimum of ≈300*psi* ±50*psi*. Gas line moisture traps are checked at least weekly and are changed or refilled as needed but will not be allowed to have less than 50% active desiccant present in the trap. Oxygen scrubbers are changed as needed.

D. A GC calibration log sheet (Appendix 19) and calibration chromatograms are maintained within each laboratory. A 1.0µl sample of a Fire Debris Calibration Mixture is injected monthly onto the non-polar, 100% methylsilicone capillary columns with a split ratio of ≈50:1. This Mixture consists of a 200:1:1 ratio of carbon disulfide, whole gasoline, and whole fuel oil #2. By adjusting the column head pressure, the 1, 2, 4-trimethylbenzene peak found in gasoline is set at a retention time of 4.0 minutes ±0.05 minute. The split flow is measured and adjusted to 50.0 *m*l/minute ±5.0 *m*l/minute. Standard, non-polar GC conditions are used. The calibration chromatograms are compared with previous ones for relative sensitivity, resolution and baseline profile.

a. If the column used for pyrolysis is NOT used for any sample injections and is only used for pyrolysis, then the fire debris calibration mixture does not need to be injected on this column. Otherwise, run the fire debris calibration mixture.

E. The polar, Carbowax column is checked similarly using a 0.10µl injection of a Polar Calibration Mixture. This Mixture consists of 80.0 *m*l of carbon disulfide, 4 drops of ethylbenzene, 4 drops of monostyrene, 8 drops of *o*-xylene, 12 drops of *m*-xylene, 8 drops of *p*-xylene, 8 drops of 1, 2, 4-trimethylbenzene, 4 drops of α-pinene, 8 drops of benzene, 8 drops of toluene, 4 drops of α-methylstyrene, 2 drops of 3-methylstyrene, 6 drops of 4-methylstyrene, 6 drops of *trans*-β- methylstyrene, 6-8 drops of furfural, 4 drops of β-pinene, 4 drops of limonene, and 24 drops of 2-butoxyethanol. The retention time of 1, 2, 4-trimethylbenzene is set to 5.0 minutes ±0.05 minute. Standard, polar GC conditions are used.

F. Record the monthly values for the retention time of 1, 2, 4-trimethylbenzene, the pressure and the split flow on a GC Calibration log sheet.

G. Monthly calibration chromatograms are maintained for each GC and kept in an appropriate file located within the Trace Evidence Section.

H. The protocol above is used whenever a new column is installed. Calibration Mixtures are prepared as needed.

I. Appropriate standards are run for all gas chromatography data and may be compared to those found in the Trace Evidence Section reference collection chromatogram books.

J. PYROPROBE UNIT

a. The pyroprobe unit is checked with a polyethylene sample in the first month of each quarter (January, April, July and October) to ensure that it continues to function. No calibration is needed at this time.

b. A piece of polyethylene plastic bag is cut to about the same sample size each time. A standard polyethylene pyrogram is then generated using standard pyrolysis conditions and non-polar GC conditions. These standard pyrograms are maintained in an appropriate file within the Trace Evidence Section.

c. The pyroprobe unit will be calibrated immediately prior to use in casework or at a minimum of once annually in January for the year prior where no pyrolysis casework has been conducted.

d. The pyroprobe quartz tube temperature is calibrated by observing the melting point of potassium chloride (≈770°C). In addition, the melting point of sodium tungstate (≈700°C) is observed within a 7*mm* melting band

on the quartz tube. These melting point temperatures are recorded on a pyrolysis log sheet (Appendix 19). The temperature of the sodium tungstate is also recorded as a part of the casework QC on the GC Conditions Sheet. Prior to calibrating the unit, the pyroprobe platinum coil is adjusted, if necessary, to achieve a uniform spacing distance between each wrap of the coil. A piece of polyethylene plastic bag is cut to about the same sample size each time and a standard polyethylene pyrogram is generated.

e. The pyrolysis unit is temperature calibrated by the manufacturer when repairs are needed.

f. The pyrolysis quartz tubes are heat cleaned at ≈1200^{o}C after each use.

g. The pyrolysis platinum coil is visually inspected before/after each use.

h. Pyroprobe o-rings are changed as needed.

♦End